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To the quest to the question of the kinetics of hydration structure formation and the properties of lime-belite binders based on marls in the karakalpakstan deposit

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Abstract— The purpose of this study was to study the kinetics of hydration structures and the physicochemical properties of lime-belite binders based on marls deposit of the Kungrad and Muynak regions of the Republic of Karakalpakstan. The experimental results show that the processes of hydration of clinker and other minerals included in the binder materials, as well as hydration structure formation (HS) in these systems are interconnected. The latter are usually a consequence of the former, so their joint consideration is of scientific and practical interest from the point of view of their role in controlling the properties of binder systems. At the same time, it was found that the formation of strength is associated not only with the formation of different structures in the system - coagulation and crystallization structures. The possibility of using (LBB) for the preparation of high-strength products of autoclave hardening is shown.

Keywords: hydration, structure formation, lime, additive, solution, binder, marl, silica, roasting, lime-belite binder.

INTRODUCTION

A fundamentally new solution to the problem of creating lime-belite materials for the construction industry of the republic is the development of an economical, fast-hardening local binder obtained from available raw materials. In this regard, it is necessary to make fuller use of local raw materials and production waste, expand the production of various products of building materials, and focus on creating new energy-saving technological processes for obtaining new lime-belite binder building materials with desired properties [1].

Lime-belite binder (LBB) obtained by low-temperature roasting of natural marls and joint grinding of the active mineral additive with lime, as well as artificial lime-silica mixtures. LBB is a finely divided mixture of materials of the proper composition. The composition may contain aluminates, ferrites and calcium aluminoferrites, at high firing temperatures, the presence of tricalcium aluminate and even tricalcium silicate is possible. (LBB) consist of a raw mixture of limestone with quartz sand, tripoli and clays. Binders as a result of hardening in autoclaves used in the production of lime-sand products are obtained using mixtures consisting of 60 ... 70% (LBB) and 40 ... 30% ground quartz sand.

The processes of hydration of clinker and other minerals that are part of the binder materials, as well as hydration structure formation (HS) in these systems are interconnected. The latter are usually a consequence of the former, so their joint consideration is of scientific and practical interest from the point of view of their role in controlling the properties of binder systems.

Material and Methods. The objects of study in this work were marl minerals from the Kungrad and Muynak deposits (Republic of Karakalpakstan). They belong to a rock of sedimentary origin, including carbonate (calcite, dolomite) and clayey (kaolinite, montmorillonite, hydromica) parts with impurities of quartz, feldspars, and so on. Carbonate rocks consist mainly of calcareous and marl minerals based on calcium carbonate $CaCO_3$. Carbonate rocks are presented as chalk-like gypsum marls and brownish-gray arganogenic-detrisus limestones.

A comprehensive study of carbonate rocks - marl minerals was carried out by the methods of X-ray phase, thermographic and physical-mechanical analysis, allowing to establish the relationship between the phase composition of the original product, the features of its hydration processes and the kinetics of formation and strength of hydration hardening structures arising on its basis.

X-ray phase analysis was used to study the structure, composition and properties of raw materials and calcined products.

Differential thermal analysis of minerals and binders was carried out on a derivatograph system F. Paulik, I. Paulik and L. Erdey. The method allows qualitatively and quantitatively to determine changes in the characteristic enthalpy, heat content of substances.

Structure formation in mineral dispersions was characterized by the kinetics of increase in plastic strength (P_m) in concentrated pastes according to the method of P.A. Rebinder, measurements were carried out on a Geppler consistometer adapted to work with a cone (axial angle 45). The load required to immerse the cone in the paste to a constant depth of at least 5 mm was determined.

It should be pointed out that the structural-mechanical properties of disperse systems can be characterized quite fully only by a complex of independent structural-mechanical constants. However, taking into account the objectives of our study and the properties of the structures under study, their fragility, we limited the number of measured parameters and, following the example, chose one parameter, plastic strength (P_m), as the structural-mechanical characteristic of the system.

These analyzes are universal and by now more modern methods for studying the compositions of solid materials.

Resullts and Discussion. In the studies, raw materials for the production of IPM were used in our case, natural marls of Kungrad and Muynak (Republic of Karakalpakstan) served.

It has been established [2] that the optimal heat treatment modes for obtaining LBB based on the studied marls are temperatures within 1100 °C with a holding time of 60 min. Under these modes, the largest amount of free calcium oxide (52...62%) and β-dicalcium silicate-belite (30...35%) is formed in the heat treatment products, in small quantities - calcium aluminates and ferrites.

The kinetics of HS can be judged from the change in the plastic strength Pm of the system, measured on a Geppler consistometer. The study of HS processes in concentrated LBB pastes makes it possible to reveal the role of the nature of the hydrated phase in the kinetics of strength formation and its creation in the emerging spatial structure.

The nature of the kinetic changes in the plastic strength of the system at a water-solid ratio W/T=0,80 (Table 1) differs from the nature of the change in the strength of the system at W/T=0,90 and 1,00. The plastic strength first increases, then, after 3 days of holding the system, it drops sharply, followed by a sharp increase again after 14 days.

The presence of drops (W/T = 0,90) in the values of plastic strength P_m is apparently associated not only with the formation of different types of structures in the system - coagulation and crystallization with the transition of the first to the second, but also with different stages of the formation of the crystallization structure [3].

Table 1.

Kinetics of structure formation P_m, MPa, in (LBB) dispersions depending on the water-to-solid ratio

LBB based on marl (deposits	W/T	Change in plastic strength P_m after exposure, days					
		1	3	7	14	28	40
Kungrad	0,80	16,7	19,5	16,8	13,8	19,7	27,9
Kungrad	0,90	14.,3	12,4	14,9	14,2	17,9	22,7
Kungrad	1,00	13,.1	12,0	13,8	13,6	16,6	20,2

Muynak	0,80	16,2	19,8	17,2	15,9	20,2	30,1
Muynak	0,90	12,5	12,8	13,8	13,9	17,9	28,8
Muynak	1,00	11,9	11,9	14,4	13,6	16,4	23,9

During the first two stages of the formation of a crystallization structure, i.e. Accordingly, the appearance of crystalline nuclei of hydrosilicates and an increase in their number without intergrowth, as well as the formation of a crystalline intergrowth of nuclei, an increase in the strength of the system occurs, which, reaching a maximum at the transition to the third stage of formation of the structure of the formation of crystalline contacts of particles, begins to weaken due to the destruction of intergrowths and recrystallization of contacts [4].

The subsequent sharp increase in the strength of the system after a minimum is due to the formation of crystalline contacts, which give the system sufficiently high strength characteristics. This area coincides in time with the 14-day exposure of the system, which is relevant to all variants of W/T [5].

The fact that the minimum extreme point on the 14 th day corresponds to the beginning of crystalline contacts is evidenced by the fact that the increase in strength after 14 days of exposure is sharp, since the strength parameters of crystallization structures, as a rule, always have relatively higher values for compared with coagulation structures, which is due to the nature of the forces that determine the contacts of particles in these structures [6]. It is known that the contacts of particles in the first (coagulation) structures are formed due to low-strength van der Waals forces of intermolecular interaction through thin interlayers of the dispersion medium and, therefore, have relatively low indices [7]. In crystallization structures, contacts are formed due to chemical forces with a much higher binding energy; they arise in the process of the formation of new phases from metastable supersaturated solutions, and these structures are intergrowth structures that differ from coagulation structures by several orders of magnitude in higher strength due to the formation of phase intergrowths.

Systems based on marl heat treatment products from the Kungradskoe deposit at W/T=0,80 under moisture-air storage conditions have a maximum strength of about 27,9 MPa, and from the Muynakskoe deposit – 30,1 MPa after 40 days of exposure.

In table. Figure 2 shows the results of physical studies of the dispersion based on the LBB. From the presented data, it can be seen that the water demand of the LBB obtained on the basis of the studied marls is greater than that of Portland cement or gypsum. This is due to the fact that the LBB composition, together with dicalcium silicate (β-C_2S), also contains large amounts of free calcium oxide CaO (58,24 and 50,30%), which, when hydrated with a large water demand, passes into calcium oxide hydrate [8].

Table 2.

The results of physical studies of pastes LBB

LBB based on marl (deposits)	Normal Density %	Setting time, min		Specific surface, cm^2/g
		Start	the end	
Kungrad	90	48	131	3200
Muynak	90	46	144	3500

In table. Figure 3 shows the dependence of the mechanical strength of LBB samples on the duration of hardening. It can be seen from the data in the table that both under the conditions of moisture-air and thermal-moist hardening, the mechanical strength of the samples increases with time, its greatest

value occurs after 28 days.

Comparison of the strength values shows that the greatest difference in their values during compression is observed in LBB based on the marl of the Kungrad deposit, which indicates a relatively higher content of silicates, aluminates and calcium ferrites in the heat treatment products of the marl of this deposit compared to the Muynak marl.

Table 3

The results of the study of the mechanical compressive strength of LBB samples in moisture-air and thermal-moist hardening.

LBB based on marl (deposit)	W/T	Compressive strength, MPa					
		moisture-air hardening, days			thermomoist hardening, days		
		3	7	28	3	7	28
Kungrad	0,80	0,96	2,81	7,31	4,02	6,38	14,16
Muynak	0,80	1,79	4,70	9,32	4,27	7,75	14,47

The initial strength of LBB is provided by the hydration of free lime and belite with the formation of the corresponding hydrates. A further very slow increase in strength (under normal conditions) is mainly due to the processes of carbonization of unbound calcium hydroxide. Therefore, with an increase in the content of the active form of SiO_2, it is possible, up to known limits, not only to intensify the hardening processes, but also to obtain a significantly higher strength of the hardened product [9].

X-ray pattern of hydrated for 28 days LBB is characterized by the presence of more intense lines of gillebrandite (0,302; 0,271; 0,260 nm), portlandite (0,486 and 0,192 nm), calcium hydrosilicate of the C-S-H type (11) (0,306 nm), as well as less intense lines of tobermorite $5CaO \times 6SiO_2 \times 5H_2O$ (0,251 and 0,248 nm). Lines 0,326, 0,205 nm and 0,186, 0,179 nm refer, respectively, to hydroaluminate and calcium hydroferrite. Activity, temperature and quenching time of LBB are given in table. 4.

Table 4.

Activity, temperature and extinguishing time of LBB

LBB based on marl (deposits	Extinguishing temperature, °C	Extinguishing time, min	Activity, %
Kungrad	43	15	70,99
Muynak	42	16	70,25

Based on the data in Table. 4, it can be assumed that LBB can be used in the production of high-strength autoclaved silicate products (cellular concrete, blocks, and others.), since the above properties of LBB correspond to the requirements for this type of product [10].

Concclusion. Thus, the versatile processes of HS in LBB dispersions obtained on the basis of marls of the Kungrad and Muynak deposits are characterized not only by the formation of different types of structures in the system - coagulation and crystallization - with the transition of the first to the second, but also by various stages of the formation of a crystallization structure. Based on the studies of the physicochemical and physico-mechanical properties of the considered LBB, it can be concluded that it is possible to obtain a highly effective binder for the preparation of high-strength autoclaved products.

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